

MECHANICAL STRENGTH DEGRADATION OF GRAPHITE FIBER REINFORCED THERMOSET COMPOSITES DUE TO POROSITY

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INTRODUCTION

The mechanical strength of composite laminates is sensitive to the presence of porosity. Porosity in laminates is generally considered to be a random distribution of voids incurred during the manufacture process. Larger, interlaminar voids typically result from trapped air or moisture; smaller, intralaminar voids may occur between fibers due to improper wetting or the release of volatile gases during the cure cycle. Porosity has its greatest effects on matrix-dominated mechanical properties such as compressive strength, transverse tensile strength and interlaminar shear strength (ILSS). Judd and Wright [1] have surveyed the existing data and made an appraisal of the effects of voids on the mechanical properties of composites. In a study of porosity in filament wound/CVD carbon-carbon composite [2], the transverse tensile strength was found to decrease exponentially with increasing porosity and followed an empirical equation often attributed to Ryshkewitch [3] and Duckworth [4]:

$$\sigma = \sigma_{\max} e^{BP} \quad (1)$$

where σ_{\max} is the strength at zero porosity, P is the volume fraction of porosity, and B (a negative number) is an empirical constant that depends on pore size, shape, and orientation. More recently Yoshida et. al. [5] took a statistical approach to the selection of appropriate criteria for porosity criticality and showed that the reduction in composite ILSS followed a linear relation with increasing porosity in the range of 0-6% by volume.

Insofar as modeling the strength reduction due to porosity, the exponent B in Eq. (1) was related to porosity characteristics such as pore size, geometry and orientation [6], but the mechanical strengths considered in such models were tensile or compressive strength of otherwise homogeneous and isotropic materials. In an anisotropic system such as composite laminates with different layup and complex porosity morphology, the modeling of the various mechanical strengths will be a major undertaking. However, simplified models do exist. For example, Greszczuk [7,8] investigated the interactions of the matrix, fiber, and

porosity from a stress concentration viewpoint and modeled the overall reduction in the composite ILSS in terms of the porosity content, fiber and matrix properties and the fiber volume fraction.

This paper describes a study of strength degradation of graphite fiber reinforced thermoset composites due to porosity. There are three objectives in this work: (1) to demonstrate that ultrasonic parameters may be used as indicators for mechanical strength by making ultrasonic NDE measurements first and then test the same pieces mechanically, (2) to obtain the phenomenological exponent B in Eq. (1) for a number of epoxy and polyimide based composite laminates to characterize the strength reduction rate, and (3) to compare the test results on unidirectional laminates with the model of Greszczuk. Due to limited availability of well-characterized specimens with known porosity data and/or ultrasonic NDE data, the number of recommended tests (e.g. in ASTM standards) was sometimes not met. However, the scattering in the data did not prevent meaningful assessment of the trends, and the emphasis is placed on the rate of strength reduction as the void content increases.

SAMPLE DESCRIPTION

Graphite/epoxy and graphite/polyimide (PMR15) continuous filament composite laminates [9] were chosen as the experimental materials due to their industrial popularity. Both thermoset composites had been consolidated into small, laminated panels from pre-preg sheets. The graphite/epoxy (gr/ep) panels were fabricated with both 16-ply unidirectional and 16-ply quasi-isotropic $(45/-45/0/90)_2$ orientation. Four unidirectional panels (A-series) and four quasi-isotropic panels (B-series) were analyzed for the correlation of porosity content to ILSS and compressive strength. Four, 8-ply gr/ep woven fabric laminates (D-series) were also included in the evaluation process. In addition, 10 graphite/polyimide (gr/PI) panels were fabricated with six plies of 8-harness woven fabric. The nominal porosity contents of both the gr/ep and gr/PI were determined by spatially averaging acid digestion results from several areas on each laminate panel. It should be noted that the gr/ep panels contained up to 6% porosity by volume while the gr/PI panels contained up to 10% porosity by volume.

Micrographic inspection was performed on representative cross-sectional slices cut perpendicular to the primary fiber orientation direction for each of the panels studied [10]. It was seen that the non-woven gr/ep laminates contained a porosity distribution consisting of elongated, elliptically cylindrical pores running parallel to the fiber axes. The axial pore length increased as the volume porosity fraction increased. On the other hand, the woven gr/ep and gr/PI contained smaller, more spherical voids located primarily in resin-rich pockets within the laminate. These voids also varied in average size with changing bulk porosity content; however, this average size did not monotonically increase with increasing porosity content as was seen in the non-woven laminates.

ULTRASONIC EVALUATION

In order to determine the feasibility in relating nondestructive ultrasonic measurements to composite laminate mechanical properties, several ultrasonic parameters were selected for correlation to mechanical strength. These characteristics, evaluated by through-transmission methods, included absolute attenuation, attenuation versus frequency slope (da/df) and longitudinal wavespeed. It has been shown that these ultrasonic properties are influenced by the porosity in composite

laminates [11-13]. Therefore, it should be possible that matrix-dominated composite properties be predicted ultrasonically due to the mutual dependence of both mechanical strength and ultrasonic measurements on the composite laminate void content. Ultrasonic transducers with center frequencies near 10 MHz were employed to determine how the aforementioned ultrasonic characteristics varied with changes in the laminate void volume fraction. Figure 1 shows the relation between these parameters and the resulting mechanical test results (presented in a later section) from areas that had been ultrasonically evaluated on a woven gr/PMR15 panel. Velocity measurements are not shown here to avoid cluttering but the correlation is also good.

MECHANICAL TEST PROCEDURES

The effects of porosity on compression strength and apparent ILSS were studied. The apparent ILSS was obtained from short-beam shear failures using a simple fixture (shown in Fig. 2) designed following the ASTM D-2344 guidelines. The compression strength was obtained on small coupons using a homemade retaining jig (also shown in Fig. 2) made of PH17-4 stainless steel for good hardness. Compression strength of porosity-free quasi-isotropic gr/ep laminates obtained with this simple fixture agreed very well with published data using elaborate fixtures like that described in ASTM D-3410.

Coupons tested in the short-beam shear fixture were approximately 1.0" long, .25" wide and .10" thick. The fixture geometry provided a span-to-depth ratio of four. Test coupons were inserted into the fixture and transversely loaded until the first major loss of load-carrying capability occurrence. This load was recorded and used to calculate the "apparent" ILSS from the relation:

$$\sigma = (3/4) \frac{P_{\max}}{A} \quad (2)$$

where P_{\max} is the maximum sustained load and A is the cross-sectional area of the coupon perpendicular to the plane of the plies. During the course of the tests, the load carried by a coupon would momentarily drop,

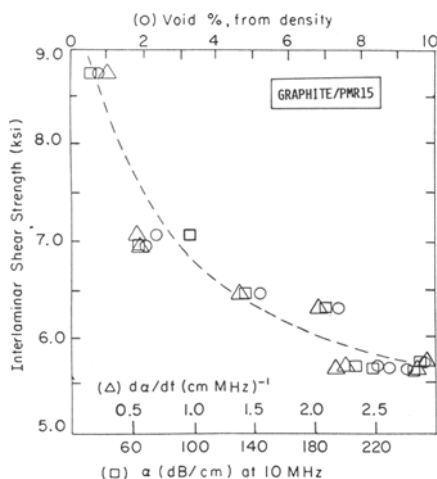


Fig. 1. Apparent interlaminar shear strength of woven graphite polyimide composite versus void content, ultrasonic attenuation and attenuation slope.

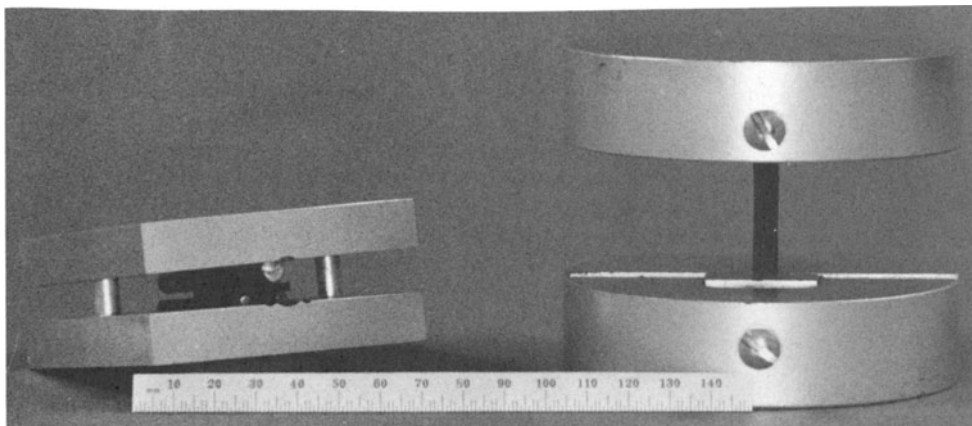


Fig. 2. Short-beam shear and compression test fixtures.

then continue to rise again. This behavior would repeat several times before a major failure occurred. This seemed to indicate that several small cracks and/or delaminations would precede the actual coupon failure. This was supported by audible cracking which preceded the failure.

Coupons for compression testing were approximately 2" long, .25" wide and .09" thick. These were placed in the testing jig and loaded in axial compression along the primary fiber orientation direction. It was seen that the amount of fixation provided at the specimen ends within the fixture had a significant effect on the compressional failure mode, particularly on the woven coupons tested. Those samples which had been clamped too loosely or too tightly generally failed within the grip area while the properly clamped samples failed cleanly at the center of the coupon gage length in a typical symmetric broom-out fashion. The compression fixture did not permit the testing of unidirectional coupons due to inadequate end restraint. Improper restraint allowed the ends to broom-out prematurely, thereby preventing acceptable failure modes characteristic of true compressive failure. The failure stress in compression was computed simply as the maximum attained axial load divided by the specimen cross-sectional area perpendicular to the load direction.

RESULTS AND DISCUSSION

Mechanical test results are shown in Figs. 3-6. Apparent inter-laminar shear strength and compressive strength are plotted against porosity volume percentages for each of the chosen laminate types. Figure 3 shows the relationship between experimentally determined short-beam-shear strength values and porosity content for the A-series gr/ep, unidirectionally reinforced coupons. Figure 4 shows the relationship between experimentally determined short-beam-shear strength values and porosity content for the D-series woven, gr/ep fabric coupons. Figure 5 shows the relationship between experimentally determined compressive strength values and porosity content for the B-series gr/ep, quasi-isotropic coupons. Figure 6 shows the relationship between experimentally determined compressive strength values and porosity content for the woven, gr/PI fabric coupons. The data in Figs. 3-5 represent averages of three tests of each composite type at each porosity level. The error bars given in Fig. 3 are also representative of the data in Figs. 4 and 5. For woven PMR-15 laminates, the limited amount

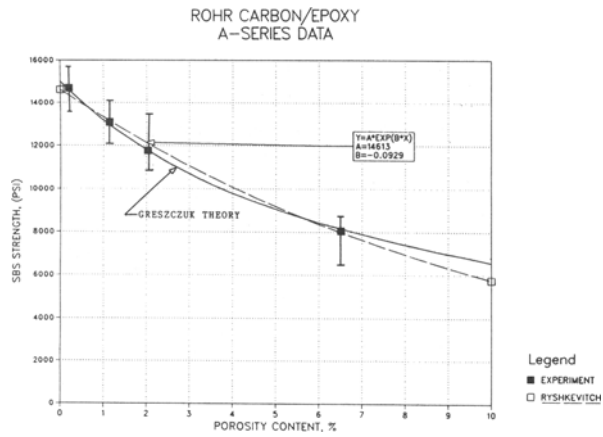


Fig. 3. Short-beam shear strength versus porosity content for unidirectional graphite epoxy laminates.

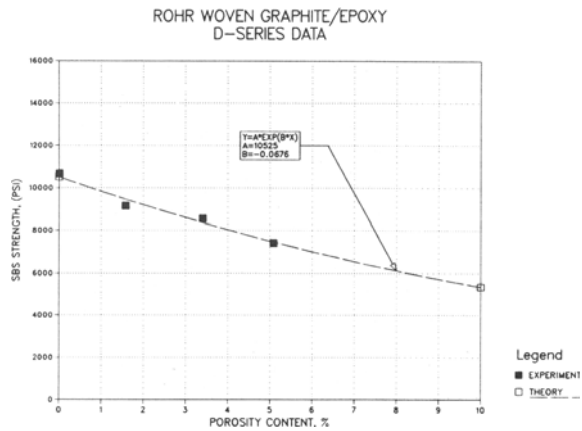


Fig. 4. Short-beam shear strength versus porosity content for woven graphite epoxy laminates.

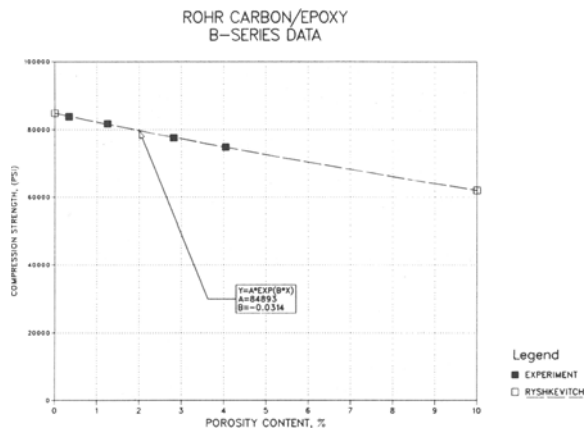


Fig. 5. Compressive strength versus porosity content for graphite epoxy quasi-isotropic laminates.

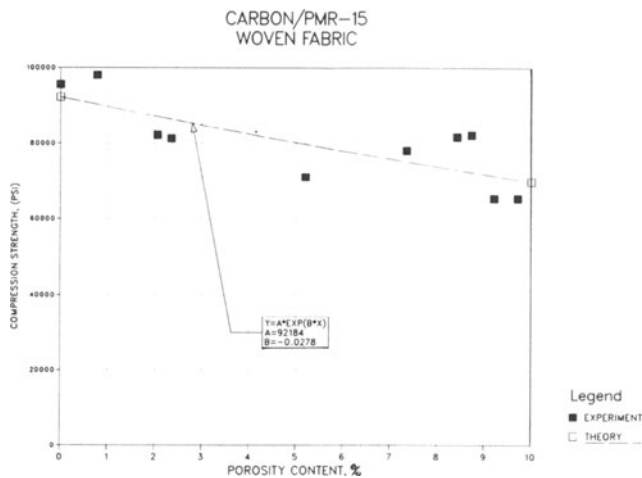


Fig. 6. Compressive strength versus porosity content for woven graphite polyimide laminates.

of specimens allowed only one test of the compression strength at each porosity level. As a result, there were considerable scatterings in the data.

In all the cases above, the mechanical strength degraded monotonically with increasing porosity content. The data were then fitted to an empirical exponential decay (Eq. (1)) to extract the value of the exponent B. In Figs. 3-6 the porosity contents are expressed as percentages, the exponent B obtained from a fit to these data should therefore be multiplied by 100 before being compared with those in Refs. 2 and 6, where the porosity contents are expressed as volume fractions. For short-beam shear strength tests, gr/ep unidirectional samples gave $B = -0.093$ and gr/ep woven laminates gave $B = -0.068$. For compression strength, the (absolute) values of B are smaller; quasi-isotropic gr/ep laminates gave $B = -0.031$ and woven gr/PI laminates gave $B = -0.028$. Empirically the range of B for a variety of homogeneous and heterogeneous solids is approximately between -3 and -9. After multiplied by 100, the values of B obtained from these mechanical strength degradation measurements fell in the same range. The data also showed that the degradation was considerably more serious for interlaminar strength than for compression strength. This is to be expected considering the location and morphology of the porosity in composite laminates.

Finally we compared the apparent interlaminar shear strength (obtained from three-point bending short-beam shear tests) of unidirectional graphite epoxy laminates with the micromechanics modeling of Greszczuk [7]. In an attempt to develop micromechanics failure criteria for components, Greszczuk considered the interaction of internal stresses due to fibers and voids and arrived at an equation (Eq. (41) in Ref. 7) for computing the shear strength of an unidirectional composite containing voids from material properties of the fiber and resin, including the resin shear strength, the fiber volume fraction, and the ratio of the fiber shear modulus to the resin shear modulus. Using best estimates of the material constants, we calculated the shear strength for unidirectional gr/ep as a function of porosity. The computed strength generally falls below the experimental value in Fig. 3 by about 20%. Considering the simplifying assumptions made, this discrepancy is surprisingly small. As a comparison of the rate of strength degradation, the Greszczuk theory was scaled to fit the experimental data and the agreement is quite good, as shown in Fig. 3.

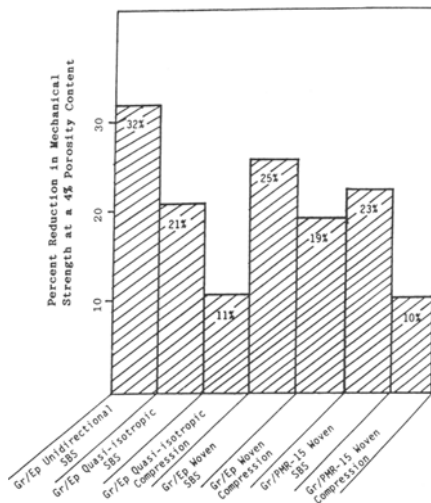


Fig. 7. Degradation of strengths of composite laminates containing 4% voids.

To compare the percent reduction of short beam shear strength and compression strength of the composite laminates tested, the results are compared in Fig. 7 for a void content of 4%. As expected, the compression strength is generally affected less than the apparent interlaminar shear strength by the same amount of porosity. Despite the limited number of specimens tested, the results agreed well with the conclusion of the Judd-Wright survey that ILSS decreases by about 28% for a 4% void content.

CONCLUSION

It was demonstrated that ultrasonic parameters may be used in monitoring the mechanical strength reduction due to the presence of porosity. The rate of strength decrease with increasing porosity was measured for shear and compression strength for a number of graphite fiber composites. For unidirectional composites, the results were also compared with a micromechanical model. The strength degradations were generally within the range of data reported in the literature.

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